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## Research Article

# Effect of Primers and Resins on the Shear Bond Strength of Resin Composite to Zirconia

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**Objective.** To evaluate the effects of various surface conditioning methods and agents. **Methods.** The intaglio zirconia substrates were air particle abraded with  $\text{Al}_2\text{O}_3$  ( $\varnothing$  50  $\mu\text{m}$ ) for 10 s. An air pressure of 450 kPa and a nozzle distance of 10 mm were used. Surface conditioning by groups: A = silane coupling agent + organophosphate adhesive; B = organophosphate primer + silane coupling agent + organophosphate adhesive; C = organophosphate primer; D = methacrylate adhesive; E = thiophosphate primer + methacrylate adhesive. Composite stubs were bonded to substrates and photo-polymerized. The specimens were thermocycled 8000 times  $55 \pm 1^\circ\text{C}$  and  $5 \pm 1^\circ\text{C}$  and kept in distilled water for 14 d. The shear bond strengths were measured with a universal testing machine. **Results.** Shear bond strengths (MPa  $\pm$  SD): Group A  $25.8 \pm 6.7$ , Group B  $26.5 \pm 8.6$ , Group C  $16.7 \pm 8.5$ , Group D  $2.6 \pm 0.7$ , and Group E  $4.2 \pm 1.2$ . ANOVA: significant differences among groups ( $P < .05$ ). Groups A and B: mainly cohesive fractures, Group C: mixed or adhesive fractures, Groups D and E: adhesive fractures. **Conclusions.** A value of 10–13 MPa is the minimum acceptable shear bond strength. Groups A, B, and C exceeded this limit, Groups D and E could not achieve the limit.

## 1. Introduction

During recent years all-ceramics, for example, yttria ( $\text{Y}_2\text{O}_3$ ) stabilized tetragonal zirconium dioxide polycrystal (Y-TZP) and aluminium trioxide ( $\text{Al}_2\text{O}_3$ ) based fixed partial dentures (FPDs), have been taken in use as substitutes for gold-alloy-based FPDs mainly because of their superior aesthetic properties and biocompatibility. Their optimal biomechanical properties [1–3], absence of metal shadowing, and elimination of possibility of metallic ion dissolution have been other arguments for their increasing use in prosthodontics as well. Nowadays, a wide range of clinical applications, for example, root canal posts, crowns, veneers, FPDs, implants, and implant abutments are available. All these applications benefit from a reliable bonding procedure which cannot be achieved by conventional etching technique used in glass-ceramic applications [4]. A value limit of 10–13 MPa is suggested as the minimum for acceptable clinical bonding [5–7].

In order to establish a durable composite resin bond to Y-TZP ceramics, many bonding procedures have been studied for several years [8]. The effects of airborne particle abrasive, tribochemical or combined treatments with or without silanizing [9] have been evaluated in numerous studies [10]. However, most of these tests should be considered short-term static studies because most have been done without any kind of fatigue cycling or exposing to the effects of water. One should keep this in mind because the primary bond to Y-TZP is micromechanical due to the inertness of the ceramic material and chemical bonding takes place between the silica-coating and the primers.

Tribochemistry involves creating chemical bonds by applying kinetic energy [11], for example, in the form of airborne particle abrasion, without any application of additional energy in the form of heat or light. Nowadays, tribochemical silicatization is a widely used and accepted pretreatment for ceramic and metal alloy restorations before cementation. It is also used for reparations in case of

ceramic fractures in FPDs. The method is sometimes referred to as a cold silicatization method as the energy needed in the silicatization process is transferred to the object material in the form of kinetic energy without any change in temperature [12, 13]. For instance, the Rocatec-system and its variation the CoJet-system (CoJet, 3M ESPE, Seefeld, Germany) are based on airborne microblasting sand, which is especially silica-modified aluminum trioxide. (Rocatec, 3M ESPE, Seefeld, Germany) The system provides the ceramic surface with a reactive silica-rich outer layer prone to the silanization and the following resin adhesion. Thus, the surface is adhesive for suitable resin composite cements.

Another silicatization method of an oxide ceramic surface is the Silicoater-technology (Silicoater, Kulzer Co., Friedrichshof, Germany) and its present day incarnation, the PyrosilPen-technology (PyrosilPen, SurA Instruments, Jena, Germany). They are based on a flame from mixture of butane gas and a silane. Silane decomposes in the flame and SiOx-C components are created. Objects put in the flame are covered by a layer of these fragments which bond adhesively to the substrate surfaces. This surface has glass-like properties and can be silanized and treated with a resin [14].

Likewise, the features of bonding resin composites containing chemical combinations capable of enhancing the bond strength to the ceramic surface have been researched in numerous studies [8, 10, 15, 16]. All the methods do not involve silica-coating or silanization as necessary part of the bonding procedure, but there are combined methods as well. Several studies suggest the use of phosphate-based, 10-methacryloyloxydecyldihydrogenphosphate-containing (10-MDP, Figure 1(a)) adhesives and cements as the best way of creating a reliable and durable bond to the oxide ceramic surface.

The aim of the present in vitro study was to evaluate the effects of some selected contemporary surface conditioning methods and the properties of various chemical combinations of resin composites and coupling agents on the shear bond strength between resin composite and Y-TZP. The hypothesis was that 10-methacryloyloxydecyldihydrogenphosphate (10-MDP) can produce significantly higher shear bond strength compared to any other bonding system evaluated in this study.

## 2. Materials and Methods

The present study was primarily designed to investigate the varying effects and capabilities of different bonding agents used in adhesion of prosthodontic restorations. First, the bonding procedures were purposely modified in order to analyze step by step the effect of presence or absence of each individual procedure and their significance on the zirconia adhesion. Secondly, in order to assess the difference of chemical bonding of a metallic surface and micromechanical bonding of a ceramic surface, some agents not originally intended to be utilized with zirconia were used. Instead of a luting cement two different particulate filled resin composites were chosen to be attached onto the conditioned zirconia surfaces.

All the materials used in this study are presented in Table 1. Forty five Y-TZP (Procera Zirconia, Nobel Biocare, Göteborg, Sweden) square-shaped specimens (2 mm × 10 mm × 10 mm) were embedded into cylinders (diameter 20 mm, height 10 mm) with an acrylic polymer material (Palapress Vario, Heraeus Kulzer, Hanau, Germany) to form the bonding substrate for the resin composite stubs. The substrate surfaces were Au sputter coated and examined with SEM (SEM, JEOL Scanning Electron Microscope JSM-5500, PGT Prism 2000, JEOL, Tokyo, Japan) as intaglio. Substrates were air particle abraded with Al<sub>2</sub>O<sub>3</sub> (Korox 50, Bego, Bremen, Germany) with a diameter of 50 µm for 10 s with a sand blasting device (CoJet, 3M ESPE, Seefeld, Germany). Air pressure of 450 kPa was used and the distance between the tip of the blasting device and the ceramic surface was 10 mm. The surfaces were subsequently examined with SEM.

The specimens were randomly divided into five study groups and the ceramic surfaces were treated as shown in Table 2. Surface conditioning was carried out as follows.

*Group A.* The surfaces were first conditioned with mixture of a silane coupling agent (Clearfil Porcelain Bond Activator, Kuraray, Osaka, Japan, Figure 1(a)) and a primer resin (SE Bond Primer, Kuraray, Osaka, Japan, Figure 1(b)) as the manufacturer recommends. The mixture was applied on the surfaces for 10 s and air-dried for 5 s. Next, a resin (SE Bond Bond, Kuraray, Osaka, Japan) was applied on the surfaces for 10 s, air-dried for 5 s and photo-polymerized for 10 s (Optilux-501, Kerr, Orange CA, USA). Wavelength maximum of the light was 495 nm and the light intensity was 550 mW/cm<sup>2</sup>.

*Group B.* The surfaces were first conditioned with an alloy primer (Alloy Primer, Kuraray, Osaka, Japan, Figures 1(b) and 1(c)) for 60 s and air-dried. Next, the surfaces were treated in the same way as in Group A.

*Group C.* The surfaces were conditioned with an alloy primer (Alloy Primer, Kuraray, Osaka, Japan, Figure 1(b)) for 60 s and air-dried. Next, a resin (Scotchbond 1, 3M ESPE, St. Paul MN, USA) was applied on the surfaces for 20 s, air-dried for 5 s and photo-polymerized for 10 s (Optilux-501, Kerr, Orange CA, USA).

*Group D.* The surfaces were conditioned with a primer (UniFil Bond Primer, GC, Tokyo, Japan, Figure 1(d)) for 20 s and air-dried for 5 s. Next, a resin (UniFil Bond Agent, GC, Tokyo, Japan) was applied on the surfaces for 10 s and photo-polymerized for 10 s (Optilux-501, Kerr, Orange CA, USA).

*Group E.* The surfaces were conditioned with a metal primer (Metalprimer II, GC, Tokyo, Japan) for 60 s and air-dried. Next, the surfaces were treated in the same way as in Group D.

The air-drying was carried out directly on the zirconia surface with gentle force and rotating movement. The

TABLE 1: Materials used in this study.

Brand	Manufacturer	Chemical composition	Batch number
Alloy Primer	Kuraray, Osaka, Japan	10-MDP methacryloyloxy-decyldihydrogenphosphate, VBATDT 6-(N-(4-vinylbenzyl)propylamino)-1,3,5-triazine-2,4-dithione <sup>1</sup>	00129B
Clearfil Porcelain Bond Activator	Kuraray, Osaka, Japan	Bis-phenol-A-polyethoxy dimethacrylate, 3-MPS methacryloxypropyl-trimethoxysilane <sup>2</sup>	00133B
Clearfil SE Bond Primer	Kuraray, Osaka, Japan	HEMA, hydrophilic dimethacrylate, 10-MDP, toluidine, camphorquinone, water, silanated silica, BisGMA	00325A
Clearfil SE Bond Bond		HEMA, hydrophilic dimethacrylate, 10-MDP, toluidine, camphorquinone <sup>3</sup>	00416A
Scotchbond 1	3M ESPE, St. Paul MN, USA	Water, ethanol, HEMA, BisGMA, dimethacrylates, silica, copolymers of itaconic and acrylic acids, photoinitiator <sup>4</sup>	3JK
Metalprimer II	GC, Tokyo, Japan	MEPS methacryloyloxyalkyl -thiophosphate derivatives, MMA methylmethacrylate <sup>5</sup>	Not available
UniFil Bond Primer	GC, Tokyo, Japan	Water, ethanol, HEMA, 4-META	0306041
UniFil Bond Agent		UDMA, TEGDMA, HEMA, fillers <sup>6</sup>	0307021
Filtek Z250	3M ESPE, St. Paul MN, USA	Bis-GMA, UDMA, TEGDMA, Bis-EMA6, silane treated ceramic <sup>7</sup>	4GP
Gradia Direct	GC, Tokyo, Japan	Dimethacrylate co-monomers, camphorquinone, UDMA, silica, fluoro alumino-silicate glass, organic filler <sup>8</sup>	0304142
Procera Zirconia	Nobel Biocare, Göteborg, Sweden	Yttria stabilized zirconium dioxide (Y-TZP) <sup>9</sup>	—
Korox 50	Bego, Bremen, Germany	Aluminium trioxide $\varnothing$ 50 $\mu\text{m}$ <sup>10</sup>	476838

<sup>1</sup>Material Safety Data Sheet ISO/DIS 11014, Printing date 09/29/2008, Reviewed on 10/01/2008, p. 1/7<sup>2</sup>Material Safety Data Sheet ISO/DIS 11014, Printing date 09/30/2008, Reviewed on 10/01/2008, p. 1/6<sup>3</sup>Material Safety Data Sheet ISO/DIS 11014, Printing date 10/02/2008, Reviewed on 10/01/2008, p. 1/6<sup>4</sup>Material Safety Data Sheet, Issue date 05/29/2008, Supersedes date 07/09/2004, p. 1/7<sup>5</sup>Material Safety Data Sheet MS 339201, Date 03/01/2006, Reviewed 01/26/2009, p. 1/2<sup>6</sup>Material Safety Data Sheet MS 000063-MS 000064, Date 01/09/2006, Reviewed 02/25/2009, p. 1/2 & p. 1/2<sup>7</sup>Material Safety Data Sheet, Issue date 05/13/2009, Supersedes date 05/08/2008, p. 1/7<sup>8</sup>Material Safety Data Sheet MS 002000, Date 01/09/2006, Reviewed 01/22/2009, p. 1/2<sup>9</sup>Conrad HJ, Seong WJ, Pesun IJ. Current ceramic materials and systems with clinical recommendations: A systematic review. J Prosthet Dent 2007; 98:389–404.<sup>10</sup>Material Safety Data Sheet, Date 02/12/1996, Reviewed 01/01/2005, p. 1/4.

TABLE 2: Ceramic surface conditioning methods and materials.

	Surface treatment	Bonding	Adherend
Group A	(1) Clearfil Porcelain Bond Activator (Kuraray) (2) Clearfil SE Bond Primer (Kuraray)	Clearfil SE Bond Bond (Kuraray)	Filtek Z250 (3M ESPE)
Group B	(1) Alloy Primer (Kuraray) (2) Clearfil Porcelain Bond Activator (Kuraray) (3) Clearfil SE Bond Primer (Kuraray)	Clearfil SE Bond Bond (Kuraray)	Filtek Z250 (3M ESPE)
Group C	Alloy Primer (Kuraray)	Scotchbond 1 (3M ESPE)	Filtek Z250 (3M ESPE)
Group D	None	(1) UniFil Bond Primer (GC) (2) UniFil Bond Agent (GC)	Gradia Direct (GC)
Group E	Metalprimer II (GC)	(1) UniFil Bond Primer (GC) (2) UniFil Bond Agent (GC)	Gradia Direct (GC)

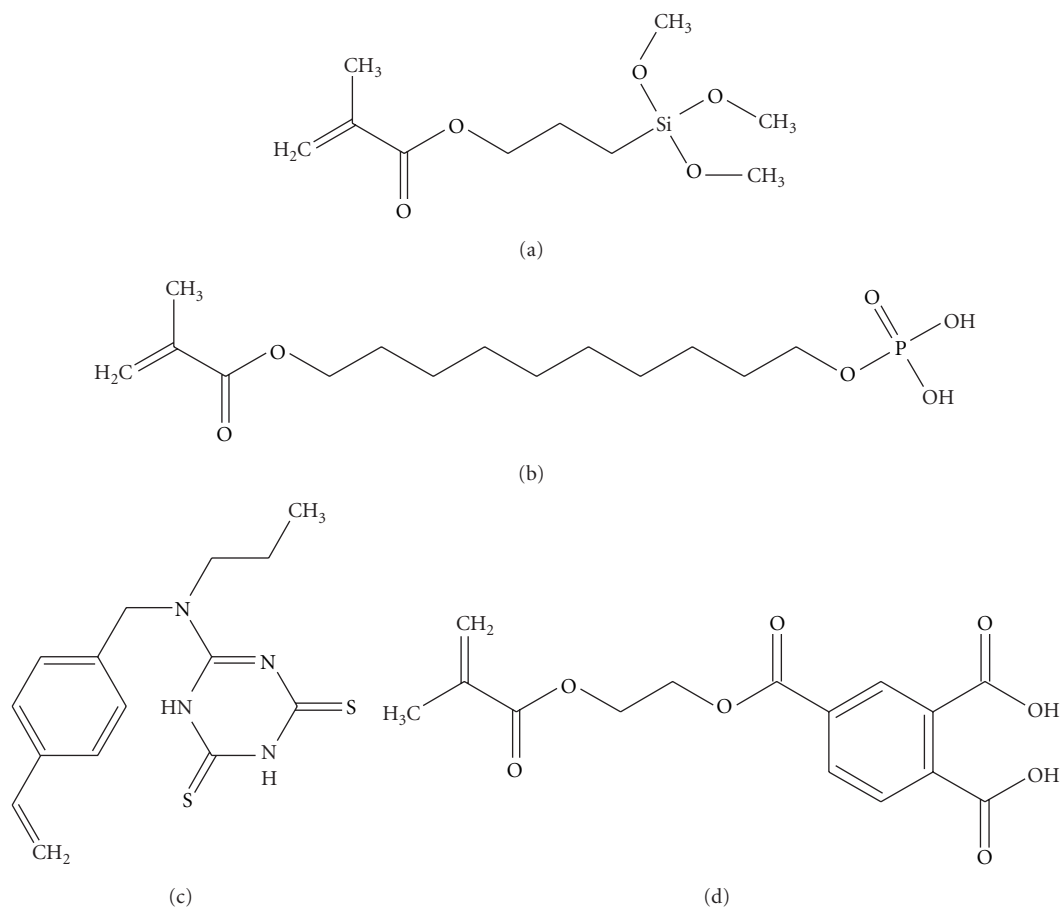


FIGURE 1: (a) 3-methacryloxypropyltrimethoxysilane (MPS). (b) 10-methacryloyloxydecyltrihydrogenphosphate (10-MDP). (c) 6-(N-(4-vinylbenzyl)propylamino)-1,3,5-triazine-2,4-dithione (VBATDT). (d) 4-methacryloyloxyethyltrimellitic acid (4-META).

curing light output was calibrated in the beginning of the preparation of each group set with the built-in radiometer of the curing unit (Optilux-501, Kerr, Orange CA, USA).

18 particulate filler resin composite stubs per group were prepared ( $n = 18/\text{group}$ ). Two stubs (diameter 3.6 mm, height 4.0 mm) were bonded to each substrate and photopolymerized for 40 s (Optilux-501, Kerr, Orange CA, USA). In Groups A, B, and C a particulate filler resin composite (Filtek Z250, 3M Espe, St. Paul MN, USA) containing *bis*-phenol A polyethylene glycol diether dimethacrylate (BISMA6), diurethane dimethacrylate *bis*-phenol A diglycidyl ether dimethacrylate (*bis*-GMA), triethylene glycol dimethacrylate (TEGDMA) and fillers was used. In Groups D and E a microfilled resin composite (Gradia Direct, GC, Tokyo, Japan) containing urethane dimethacrylate (UDMA), dimethacrylate and fillers was used.

Thermocycling was used as a custom artificial ageing method. The specimens were thermocycled 8000 times between  $55 \pm 1^\circ\text{C}$  and  $5 \pm 1^\circ\text{C}$  (Thermocycling unit 8l, custom-made, Biomechanical Testing Laboratory, University of Turku, Turku, Finland). Duration of exposure to both temperatures was 20 s at a time unintermittently. The specimens were kept in distilled water at  $37^\circ\text{C}$  for 14 d.

The shear bond strengths of the composite to Y-TZP were measured using a universal testing machine with a cross-head speed of 1.0 mm/min (Lloyd Instruments LRX Material Testing Machine, Lloyd Instruments, Fareham, England) with a parallel knife-edge blade touching the interface of the ceramic and resin composite cylinder (Figure 2).

Fracture surfaces were examined with a scanning electron microscope (SEM, JEOL Scanning Electron Microscope JSM-5500, PGT Prism 2000, JEOL, Tokyo, Japan). The failure type of debonding was defined as follows: when about 2/3 or more of the resin composite cement was left on the ceramic surface, the failure was designated as “cohesive,” and when less than 1/3, “adhesive,” respectively. Failure mode was designated as “mixed,” when it fell between the limits above.

Two-way analysis of variance (ANOVA) followed by Tukey post-hoc test and Weibull analysis were used as statistical methods. Weibull distribution has been designed to compute failure probabilities as a function of applied stress. It has been used to characterize the nature of the shear bond strength by fitting the statistical distribution to life data in order to estimate the failure probability.





FIGURE 2: The specimen in the shear bond strength test.

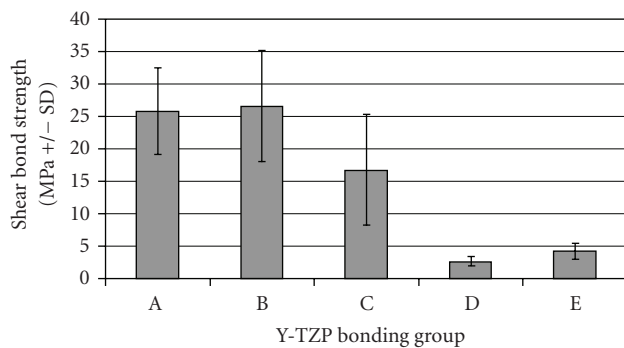


FIGURE 3: Mean values of shear bond strength (MPa) of each group with standard deviations. Group A<sup>a</sup> = silane with organophosphate adhesive. Group B<sup>a</sup> = organophosphate primer with silane and organophosphate adhesive. Group C<sup>a</sup> = organophosphate primer. Group D<sup>b</sup> = methacrylate adhesive. Group E<sup>b</sup> = thiophosphate primer with methacrylate adhesive. Superscript letters represent statistically different groups (Tukey,  $P < .05$ ).

### 3. Results

The mean values of data of shear bond strengths are presented as a bar diagram in Figure 3. The characteristic Weibull strengths are presented in Figure 4.

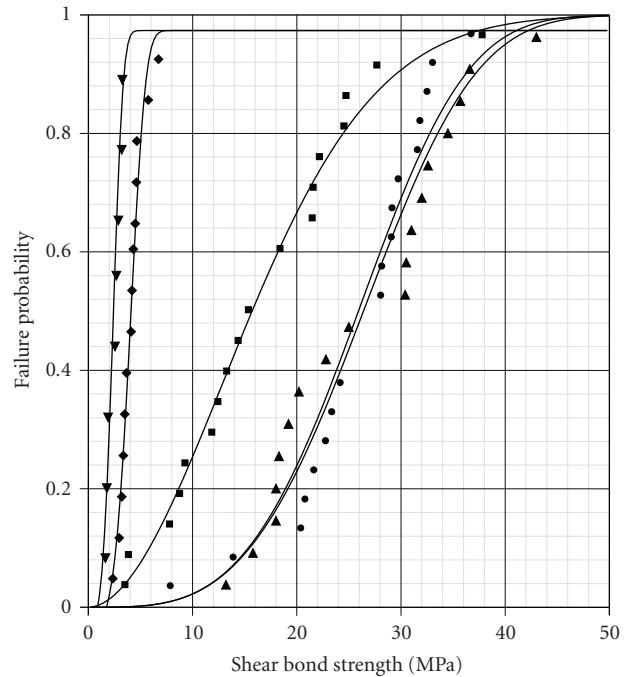
Statistical analysis (ANOVA) showed significant difference among groups ( $P < .05$ ).

Groups A and B showed improved shear bond strength values whereas the results in Group C were considerably lower. However, Groups D and E could show only very low shear bond strengths.

In Groups A and B the fractures occurred mainly within composite resin and thus were cohesive or mixed. In Group C the fractures were either mixed or adhesive whereas in Groups D and E the fractures were solely adhesive. The fracture types by groups are presented in Table 3.

TABLE 3: The number of each fracture type in each group.

	Cohesive	Mixed	Adhesive
Group A	15	1	2
Group B	13	2	3
Group C	6	1	11
Group D	0	0	18
Group E	0	0	18



Multi-plot

- A,  $\beta_1 = 3.59$ ,  $\eta_1 = 28.7$ ,  $\rho = 0.946$
- ▲— B,  $\beta_2 = 3.52$ ,  $\eta_2 = 29.3$ ,  $\rho = 0.97$
- C,  $\beta_3 = 1.9$ ,  $\eta_3 = 19$ ,  $\rho = 0.988$
- ▼— D,  $\beta_4 = 3.82$ ,  $\eta_4 = 2.8$ ,  $\rho = 0.961$
- ◆— E,  $\beta_5 = 4.15$ ,  $\eta_5 = 4.6$ ,  $\rho = 0.976$

FIGURE 4: Weibull graphs of shear bond strengths of composite resins to zirconia as a function of various surface conditioning methods.

### 4. Discussion

The present study design was originally created in order to compare the effects and possible synergetic benefits of some common, ripe, and compatible bonding agents used in everyday practice. The individual bonding agents and other materials were handled according to the manufacturers' recommendations although the procedures themselves were modified and the primers were applied on purpose on "incorrect" purpose. The aim was to standardize the application, air-drying, light-curing, and testing procedures as coherent as possible but the operator's effect on the results must be kept in mind. All the samples were prepared by the same operator but the procedure itself involves so-called human factor, in the form of possible inaccuracy, for example, in the amount of bonding agent applied, the

proportioning of air boost or the light-curing angle. All these might be sources for random or systematic errors.

The other factor affecting the results is the artificial fatigue method selected. In numerous studies thermocycling and varying times of water storage have been used as an artificial ageing method [17–21]. It is a well-known fact that thermocycling is a controversial method and some other testing methods have been suggested [22]. However, thermocycling is in accordance with the ISO 10477 standard concerning the ageing of a bond [23]. It is noteworthy that there is no concrete evidence that failures in practice would occur because of thermal stresses, notwithstanding the theoretical expectation. However, the distinction must be made between the equivalent static stress test and fatigue failure, where repeated loading to a stress below the static strength occurs. Also, it has never been determined whether it is the time at temperature, that is, cumulative duration under stress, as opposed to true fatigue, that is relevant factor. In other words, whether failure occurs due to flow, that is, deformation, in one or other of the layers in the bonded structure, is unknown. This is obviously dependent on the glass transition temperatures ( $T_g$ ) of these bonded components which have not been determined. Such flow would lead to collapse in a truly static test at a stress below the ordinary static strength. Cycling testing as such cannot discriminate these issues, and ignoring this might account, in part, for the inconsistency of test results so far reported. The test outcome depends on the stress generated and the failure mechanism [24]. However, due to the mechanism of the failure caused by cycling testing it is necessary to consider other kind of fatigue tests that can be more suitable for clarifying the nature of the failure and the factors leading into it.

Some previously published *in vitro* studies concerning bonding to alumina or zirconia ceramics have been dealing with either the effect of the presence of silica coating achieved with various methods [8, 14, 17] or the features of different luting cements [16, 25]. Different surface pretreatment methods [26] of the ceramic substrate have been discussed widely as well [8, 17]. As the results of the present study suggest, the surface treatment procedure has a considerable influence on bond strength to ceramic material. 10-MDP-containing materials together with silanization had a superior ability to form a stabile bond. On the one hand, high shear bond strength is a result from the capability of silane to wet the surface of a substrate lowering thus the surface tension. Hydrophilic silane coupling agents make surface energy higher and hence can promote the interaction between zirconia ceramics and resin composite cements and form covalent bonding [27]. Thus, hydrophobic matrix (resin composite) can adhere to hydrophilic surfaces, such as silica, glass, glass-ceramics, and, with adequate pretreatment, even Y-TZP. One recently presented theory concerning silane coupling agents suggests that the silanes in some way modify the oxide layer of the substrate and thus promote adhesion [28]. It is of further importance to study the mechanism and the actual meaning of the silane on the shear bond strength which today still is not completely understood. It must be remembered that there is a wide variety of different silane monomers (i.e., methacrylate, acrylate) and

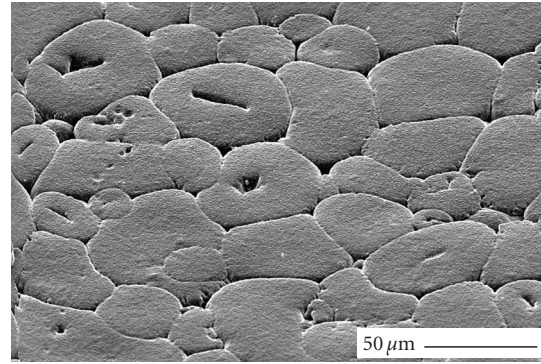


FIGURE 5: Tilted SEM image of intaglio surface of Procera Zirconia.

their functionality may affect the bond strength as well [29].

On the other hand, in the other end of MDP-molecule there is a reactive carbon-carbon double-bond that enables its effective and durable polymerization to the resin composite. More importantly, the phosphate ester group of the adhesive monomer MDP may also bond directly to metal oxide surfaces and ceramics, such as zirconia, and can form a hydrolytically stable bond. Therefore, some former studies [8, 10, 30, 31] and the present results suggest a chemical bond at some extent between MDP and Y-TZP.

Combinations without MDP could not form an acceptable bond to Y-TZP according to the present study. Formerly has been discovered [32] that the phosphoric primer is effective in the bonding of luting agents to cast pure titanium, and further, that a primer containing methacryloyloxy-alkylthiophosphate (MEPS) is also reactive and promotes adhesion. However, the present study cannot suggest the use of MEPS in zirconia ceramics bonding as there are more effective primers and procedures to achieve an acceptable bond strength level.

SEM imaging revealed that relatively high air pressure used initially in airborne particle abrasion procedure caused visible increased surface roughness (Figures 5, 6, and 7). This can be explained by comparatively high kinetic energy and by the fact that even though zirconia is hard, it is at the same time a relatively ductile material [33, 34]. Increased surface roughness improves shear bond strength by providing more sites for microretention and by increasing the substrate surface area of the ceramic surface for bonding. Additionally, airborne particle abrasion removes the possible impurities, such as oil, grease, and other contaminants, from the substrate surface to be bonded.

The shear bond strengths produced by using the alloy primer, resin primer, and resin containing 10-MDP (10-methacryloyloxydecyl dihydrogenphosphate, Figure 1(b)) were significantly higher than without. The polymers and the compounds of the adhering particulate filler resin composite did not have the bonding capacity.

The bonding capability of metal primer, resin primer and resin containing MEPS and 4-META (4-methacryloyloxyethyl trimellitate anhydride, Figure 1(d)) were considerably inferior achieving only poor shear

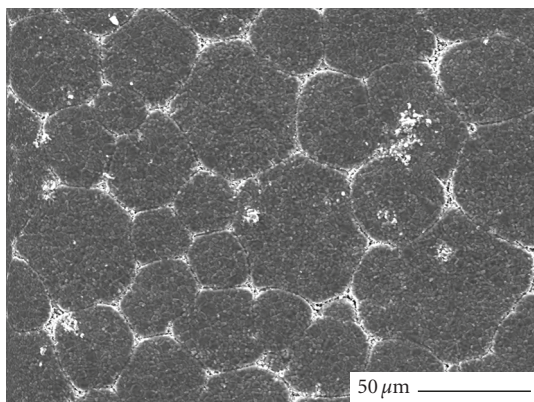


FIGURE 6: SEM image of intaglio surface of Procera Zirconia.

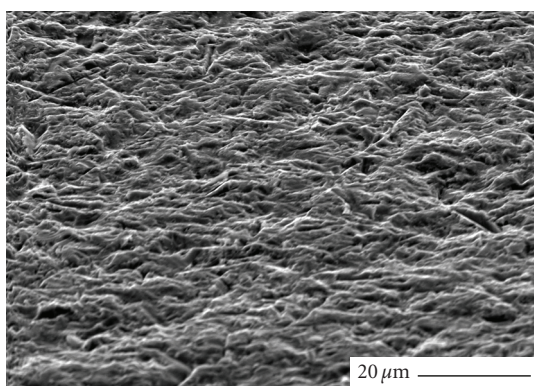


FIGURE 7: SEM image of air particle abraded surface of Procera Zirconia.

bond strength values. The adhering particulate filler resin composite used in Groups D and E contained partially the same compounds as the composite in Groups A, B, and C and did not have the bonding capacity.

Thereby, superior shear bond strength values when the following three compounds were used.

- (1) Metal alloy primer (Alloy Primer, Kuraray, Osaka, Japan) containing 10-methacryloyloxydecyldihydrogenphosphate (10-MDP, Figure 1(b)) and 6-(N-(4-vinylbenzyl)propylamino)-1,3,5-triazine-2,4-dithione (VBATDT, Figure 1(c)).
- (2) Silane (Clearfil, Porcelain Bond Activator, Kuraray, Osaka, Japan) containing 3-methacryloxypropyltrimethoxysilane (MPS, Figure 1(a)) together with resin primer (SE Bond Primer, Kuraray, Osaka, Japan) containing 10-MDP (Figure 1(b)).
- (3) Resin (SE Bond Bond, Kuraray, Osaka, Japan) containing 10-MDP (Figure 1(b)).

From the clinical point of view, ceramic restorations are often utilized in minimal invasion techniques. Tooth cavity preparation does not always provide sufficiently mechanical retention due to the anatomy of the tooth, and therefore the importance of proper wetting followed by adhesive bonding

cannot be overlooked. A shear bond strength limit of 10–13 MPa has been suggested as the minimum for acceptable clinical bonding [5–7] and almost every system assessed in this study with its short-term static tests exceeds this limit in. The limit proposed by ISO 10477 standard is merely 5 MPa [23]. However, the results may be different, inferior, when extended thermocycling time is applied [29]. Successful long-term bonding requires proper knowledge over each individual adhesive material and control over pre-treatment techniques as well as meticulous working. The most important individual factor in order to achieve the highest possible shear bond strength is selecting a reliable bonding system. Such system includes a silane coupling agent, both a primer and an adhesive resin containing 10-MDP as well as a fatigue resistant resin cement. In the present study Groups A and B clearly exceeded the acceptable bond limit [5–7].

## 5. Conclusions

Based on the results the following conclusions might be drawn.

- (1) The presence of 10-methacryloyloxydecyldihydrogenphosphate in the surface treatment agents or in the bonding agents increased considerably the shear bond strength between the ceramic material and resin composite.
- (2) 10-methacryloyloxydecyldihydrogenphosphate only in an alloy primer applied onto the ceramic surface did not produce as high shear bond strength values as by using both resin primer and the adhesive bonding resin containing 10-methacryloyloxydecyldihydrogenphosphate (10-MDP).

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